

## CLAIMS

1. A process for the deprotection of an AcM-, MBzl- and/or tBu-protected thiol which comprises reacting said protected  
5 thiol with an acid in the presence of an oxidising agent at a temperature sufficient to effect deprotection and generation of disulphide bonds.

10 2. A process as claimed in claim 1 wherein said acid is trifluoroacetic acid.

3. A process as claimed in either one of claims 1 or 2 wherein said oxidising agent is DMSO.

15 4. A process as claimed in any one of claims 1 to 3 wherein deprotection is effected using a TFA/DMSO mixture comprising 1 to 20% DMSO.

20 5. A process as claimed in any one of claims 1 to 4 wherein said protected thiol is present in a peptide.

25 6. A process as claimed in claim 5 wherein said peptide comprises at least two tBu protected thiols and/or at least two AcM or MBzl protected thiols.

7. A process as claimed in any one of claims 1 to 6 wherein tBu protected thiols are deprotected at room temperature.

30 8. A process as claimed in any one of claims 1 to 7 wherein AcM or MBzl protected thiols are deprotected at temperatures of 30°C or above.

35 9. A process as claimed in claim 8 wherein AcM or MBzl protected thiols are deprotected at temperatures of 50°C or above.